

Crystal and Molecular Structure of Sarcosine

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The crystal structure of sarcosine in pure form has been determined at low temperature (135 K). The crystals are orthorhombic, space group $P2_12_12_1$ with unit cell dimensions $a = 6.687(2)$ Å, $b = 7.899(3)$ Å, $c = 8.607(3)$ Å. On the basis of 988 reflections with intensities larger than $3\sigma(I)$ the structure was refined to a conventional R -factor of 0.034 to give e.s.d. in bond lengths of 0.002 Å and 0.2° in angles. The molecule is extended and exists in the zwitterionic form. There is a small deviation from planarity in the α -amino acid group as well as in the main chain.

Sarcosine (N -methylglycine, $\text{CH}_3\text{NH}_2^+\text{CH}_2\text{COO}^-$) is an α -amino acid found to be present in several biologically important compounds and is also used in certain cosmetics.^{1,2} Although structural studies have already been made on several derivatives of this amino acid,^{2–6} there appear to be no studies of the compound itself. Accordingly we have carried out the crystal structure analysis of sarcosine in its pure form at low temperature. The analysis is also of interest for the study of crystal effects on the conformation of this compound.

Experimental

The crystal used for the X-ray experiment was found in a commercial sample from SIGMA. The experimental data is given in Table 1. Three test reflections were measured at intervals of 135 reflections during the intensity data collection. No loss of intensity was found. Corrections were made for Lorentz and polarization effects and the unit cell dimensions were determined from diffractometer setting angles for 20 reflections. Standard deviations in the measured intensities were calculated as $\sigma(I) = [C_T + (0.02C_N)^2]^{1/2}$, where C_T is the total number of counts and C_N is the scan count minus the background count. The coordinates of all non-hydrogen atoms were determined by direct methods.⁷ Refinements were performed by least-squares calculation; hydrogen atom positions were calculated and included in the refinements. The least-squares calculations proceeded with anisotropic temperature factors for the non-hydrogen atoms and isotropic temperature factors for the hydrogen atoms. Computer programs used are described in Refs. 8 and 9. Positional parameters are given in Table 2; lists of anisotropic thermal parameters and structure factors may be obtained from the authors on request.

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Description and discussion

The sarcosine molecule is illustrated in Fig. 1 where the numbering of the atoms and bond lengths and angles are given. The molecule is in an extended form and exists in the zwitterionic state as two hydrogen atoms are located at the nitrogen atom. The molecular main chain is not quite pla-

Table 1. Crystal and experimental data.

Compound	$\text{C}_3\text{O}_2\text{NH}_7$
Melting point/°C	213
Diffractometer	NICOLET P3/F
Crystal size/mm	0.45×0.45×0.25
Radiation	Graphite cryst. monochrom. MoK_α ($\lambda = 0.71069$ Å)
Crystal system	Orthorhombic.
$a/\text{Å}$	6.687(2)
$b/\text{Å}$	7.899(3)
$c/\text{Å}$	8.607(3)
$V/\text{Å}^3$	454.6(3)
Temp./K	135
Space group	$P2_12_12_1$ (No. 19)
M/D	89.095
Z	4
$F(000)$	192
$D_x/g \text{ cm}^{-3}$	1.301
μ (Mo- K_α)/ cm^{-1}	1.0
Scan mode	$\theta/2\theta$
Scan speed (2θ)/° min^{-1}	6.0
Scan range (2θ)/°	$2\theta_{\alpha_1} - 1.0$ to $2\theta_{\alpha_2} + 1.1$
Maximum ($\sin\theta/\lambda$)/ Å^{-1}	0.81
No. of independent measurements	1024
No. with $I > 3\sigma(I)$	988
Correction for absorption	no
Method to solve structure	MITHRIL
No. of parameters refined	83
$R = \sum F_o - F_c / \sum F_o$	0.034
$R_w^a = [\sum w(F_o - F_c)^2 / \sum w F_o^2]^{1/2}$	0.040
$S = [\sum w(F_o - F_c)^2 / (n - m)]^{1/2}$	2.21

^a w is the inverse of the variance of the observed structure factors.

Table 2. Fractional atomic coordinates of sarcosine. Estimated standard deviations in parentheses. $U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3$.

Atom	x	y	z	U_{eq}
O1	0.2189(2)	0.1810(1)	0.1719(1)	0.019
O2	0.2329(2)	-0.0930(1)	0.2370(1)	0.025
N1	-0.0153(2)	-0.0622(1)	0.4798(1)	0.014
C1	0.1765(2)	0.0548(2)	0.2572(2)	0.016
C2	0.0448(2)	0.0943(2)	0.3973(2)	0.016
C3	-0.1801(2)	-0.0339(2)	0.5923(2)	0.023
H11	0.089(3)	-0.101(3)	0.541(2)	0.030
H12	-0.056(3)	-0.142(2)	0.413(2)	0.022
H21	0.112(3)	0.163(2)	0.472(2)	0.022
H22	-0.076(2)	0.151(2)	0.367(2)	0.014
H31	-0.200(3)	-0.143(3)	0.651(2)	0.027
H32	-0.298(3)	-0.001(2)	0.538(2)	0.035
H33	-0.134(3)	0.059(3)	0.669(3)	0.033

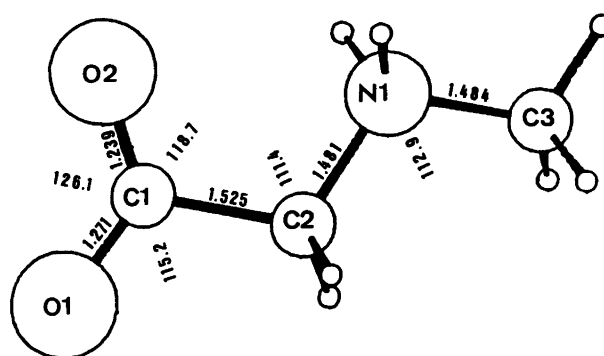


Fig. 1. The sarcosine molecule. Bond lengths and angles are indicated. The e.s.d. are 0.002 Å in bond lengths and 0.2° in angles. The C-H bonds are all in the range 0.95–1.03 Å and the N-H bonds are between 0.90 and 0.93 Å.

nar in the crystal as the torsional angle about the C2–N1 bond (C1–C2–N1–C3) is $-166.3(2)^\circ$ and the carboxy group is rotated 6° about the C2–C2 bond [O1–C1–C2–N1: $173.7(2)^\circ$ and O2–C1–C2–N1: $-6.8(2)^\circ$]. The packing of the molecules in the crystal is illustrated in Fig. 2 where the hydrogen bonding is also indicated. The geometry of the hydrogen bonding is given in Table 3. It may be seen from the stereo plot that the nitrogen atom acts as a donor in two hydrogen bonds, both involving the O1 atom. Thus each molecule is connected to four neighbouring molecules to form hydrogen-bonded layers perpendicular to the x -axis. Between these layers there are only van der Waals forces and the shortest contacts are found between the N -methyl

group in one layer and the carboxylic oxygens in another (O1...H33: 2.66 Å, O2...H31: 2.34 Å). Even if the molecule exists in the zwitterionic state, it is notable that the C1–O1 bond is significantly longer than the C1–O2 bond. It seems reasonable to explain this by the fact that the O1 atom is engaged in two hydrogen bonds whereas O2 is not involved in such bonds. A similar bond length difference in the sarcosine carboxylic group is found in the sarcosine telluric acid adduct⁶ where one of the carboxylic-group oxygens in one of the two sarcosine molecules is involved in three hydrogen bonds and the other oxygen atom is engaged in only one. The two other structure analyses of sarcosine derivatives^{5,6} reporting distances and angles with

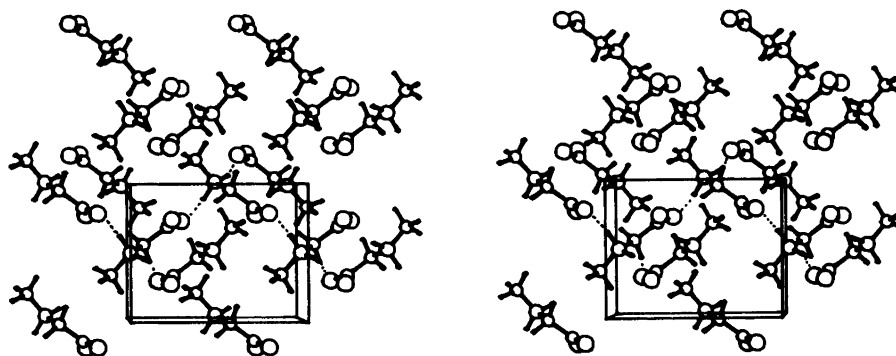


Fig. 2. Stereoscopic drawing of the packing of sarcosine molecules in the crystal as seen down the y -axis. The hydrogen bonding is indicated.

Table 3. The geometry of the hydrogen bonds in sarcosine crystals.

A	D	A...D	A...H	C–A...H	A...H–D
O1	N1 (1/2– x , – y , –1/2+ z)	2.746 Å	1.823 Å	104.0°	176.1°
O1	N1 (– x , 1/2+ y , 1/2– z)	2.770 Å	1.919 Å	131.6°	158.2°

Table 4. Comparison of the geometry of sarcosine molecules from different structure analyses.

	sarc. CaCl ₂ ⁵ (T = 118 K)			(sarc) ₂ Te(OH) ₆ ⁶ (room temp.)		Sarcosine (T = 135 K)
O1-C1	1.253(1)	1.240(3)	1.249(3)	1.252(4)	1.266(4)	1.271(2)
O2-C1	1.247(1)	1.250(3)	1.254(3)	1.254(4)	1.238(4)	1.239(2)
C1-C2	1.526(2)	1.521(3)	1.518(3)	1.515(5)	1.522(5)	1.525(2)
C2-N1	1.483(1)	1.493(3)	1.479(3)	1.483(4)	1.484(5)	1.481(2)
N1-C3	1.487(2)	1.474(4)	1.502(4)	1.485(4)	1.476(5)	1.484(2)
O1-C1-O2	128.2(1)	128.5(2)	127.0(2)	126.5(3)	126.4(3)	126.1(2)
O1-C1-C2	114.5(1)	113.9(2)	115.7(2)	117.5(3)	115.2(3)	115.2(2)
O2-C1-C2	117.3(1)	117.5(2)	117.3(2)	116.0(3)	118.3(3)	118.7(2)
C1-C2-N	111.8(1)	112.5(2)	111.6(2)	115.3(3)	112.9(3)	111.4(2)
C2-N-C3	111.9(1)	112.6(2)	112.4(2)	112.1(3)	114.0(3)	112.9(2)
O1-C1-C2-N1	-178.8	-177.3	176.6	-165.3	167.6	173.7
O2-C1-C2-N1	1.5	8.1	3.9	14.0	-14.8	-6.8
C1-C2-N1-C3	-179.7	-175.1	177.5	172.1	-83.1	-166.3

an accuracy comparable to those found in the present work represent five independent sarcosine molecules in zwitterionic form. A comparison of the geometry of the sarcosine molecule from these structure analyses together with the present results, is given in Table 4. It may be noted that all the molecules except one are found to have an C1-C2-N1-C3 antiperiplanar conformation whereas one of the two independent molecules in the telluric acid adduct exists in a synclinal conformation. However, the differences in conformation do not appear to have any significant influence on the distances and angles.

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